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Evidence for melting mud in Earth's mantle from extreme oxygen isotope signatures in zircon

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ABSTRACT

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Melting of subducted sediment remains controversial, as direct observation of sediment melt generation at mantle depths is not possible. Geochemical fingerprints provide indirect evidence for subduction-delivery of sediment to the mantle, however sediment abundance in mantle-derived melt is generally low (0%–2%), and difficult to detect. Here we provide evidence for melting of subducted sediment in granite sampled from an exhumed mantle section. Peraluminous granite dikes that intrude peridotite in the Oman-United Arab Emirates ophiolite have U-Pb ages of 99.8 ± 3.3 Ma that predate obduction. The dikes have unusually high oxygen isotope ($\delta^{18}O$) values for whole rock (14–23‰) and quartz (20–22‰), and yield the highest $\delta^{18}O$ zircon values known (14–28‰; values relative to VSMOW). The extremely high oxygen isotope ratios uniquely identify the melt source as high $\delta^{18}O$ marine sediment (pelitic and/or siliciceous mud), as no other source could produce granite with such anomalously high $\delta^{18}O$. Formation of high $\delta^{18}O$ sediment-derived (S-type) granite within peridotite requires subduction of sediment to the mantle, where it melted and intruded overlying mantle wedge. The granite suite described here contains the highest oxygen isotope ratios reported for igneous rocks, yet intruded mantle peridotite below the Mohorovičić seismic discontinuity, the most primitive oxygen isotope reservoir in the silicate Earth. Identifying the presence and quantifying the extent of sediment melting within the mantle has important implications for understanding subduction recycling of supracrustal material and effects of on mantle heterogeneity over time.

INTRODUCTION

Subduction of lithosphere to the upper mantle is a fundamental tenet of plate tectonics (Wilson, 1963). Detection of sediment contributions to mantle-derived melt

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provides evidence for transport of sediment to the mantle via subduction. Geochemical tracers used to detect the presence of sediment in mantle melt (i.e., basalt) include trace elements (Ba, La, Th, and others, e.g., Plank and Langmuir, 1993), and isotopes ($\delta^{18}O$, $\delta^{37}Cl$, ^{10}Be, and others, e.g., Eiler et al., 1995; John et al., 2010; Dreyer et al., 2010). Calculated sediment abundances in basalt are typically low, <2% (e.g., Eiler et al., 1995). Here, field observations, petrologic data, and oxygen isotope evidence are used to show that peraluminous granite dikes intruding peridotite in the Oman-United Arab Emirates (UAE) ophiolite formed by melting of subducted marine sediment in the mantle.

Opportunities to observe in situ melt generated in the mantle are rare, and generally restricted to ophiolites, sections of oceanic crust and mantle exposed at convergent plate margins (Dewey, 1976). The Oman-UAE ophiolite is considered the type-example of an ophiolite, consisting of a section of oceanic crust and mantle that was obducted onto the Arabian continental margin in the Late Cretaceous (Fig. 1A; Searle et al., 2015). The crustal section includes ~8 km of pillow basalt, sheeted dikes, and gabbro above the crust-mantle transition, or petrologic Moho. The underlying mantle section consists of ~15 km of peridotite (harzburgite), and hosts the dike suite studied here (Fig. 1B).

Three distinct suites of granites (*sensu latu*) are recognized in the Oman-UAE ophiolite. Granites intruding the crustal section form a 'crustal plagiogranite' suite that ranges from granodiorite to tonalite, has SiO_2 from 54 to 72 wt.%, and low K_2O (<1 wt.%). The crustal plagiogranite suite is metaluminous, contains zircon with $\delta^{18}O$ from ~4–5‰, and formed by fractionation of mantle melt or anatexis of oceanic crust (Grimes et al., 2013; Rollinson, 2015). Two additional suites intrude peridotite; one has SiO_2 from

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67%–75%, is metaluminous, and ranges from granodiorite to tonalite. The other suite intrusive to the mantle and the focus of this study has higher SiO_2 values (72%–78%), it is peraluminous and is predominately true granite (*sensu stricto)*, consisting of near-equal proportions of quartz, plagioclase, and alkali feldspar (Cox et al., 1999; Rollinson, 2015; Searle et al., 2015). The origin of the peraluminous suite is debated; proposed formation mechanisms include fractionation from mafic melt (Peters and Kamber, 1994), anatexis of pelitic material (Cox et al., 1999; Searle et al., 2015), and mixtures of melted crust and sediment (Rollinson, 2015; Haase et al., 2015). A key observation recognized from field studies (Peters and Kamber, 1994) and confirmed by zircon U/Pb dating (Styles et al., 2006; Rioux et al., 2013; this study) is that granite in the mantle section formed synchronous with ocean crust formation at ca. 99 Ma, and thus pre-date ophiolite obduction from ca. 85–90 Ma
(Searle et al., 2015; Styles et al., 2006). The pre-obduction intrusive relationship distinguishes the Oman-UAE granite suite from other ophiolites where granite intruded peridotite post-obduction (e.g., Ronda peridotite, Priem et al., 1979).

METHODS AND SAMPLES

We present $\delta^{18}O$, U-Pb, trace element, and εHf data for zircon, and $\delta^{18}O$ for whole rock and quartz from ten dike samples (see the GSA data repository[1] item DR1 for methods). Zircon grains were imaged by scanning electron microscopy, analyzed for oxygen isotope ratio using secondary ion mass spectrometry (SIMS), and U-Pb age, trace element, and εHf by laser ablation inductively couple plasma mass spectrometry (LA-

ICPMS) (Items DR1, DR2). Analyses of whole rock and quartz for oxygen isotope ratio were made by laser fluorination (Items DR1, DR2).

Ten peraluminous granite dikes along a ~250 km length of the mantle section of the Oman-UAE ophiolite were analyzed (Fig. 1; Item DR1). The dikes range from 2 to 8 m in width (Fig. 1B). Major and trace element data for dikes at each of the three sampling sites (our samples with prefixes 13- and 14-) were reported previously (Rollinson, 2015).

The granite dikes are uniformly peraluminous (Al_2O_3/Na_2O+K_2O+CaO >0.99), high in SiO_2 (~74%–77% wt.%), generally low in Fe, Mg, Ca, and Na, and all but one has normative corundum (0.4%–0.9%) (Item DR1). The peraluminous granites contain accessory minerals such as muscovite, biotite, garnet, cordierite, andalusite, lepidolite, tourmaline, and zircon (Rollinson, 2015; Item DR1). Some samples exhibit evidence of alteration of various minerals to secondary phases.

RESULTS

Zircon U-Pb, trace elements, and εHf by LA-ICPMS

Zircon grains from seven five samples were analyzed for U/Pb, targeting both cores and rims and yield a $^{206}Pb/^{238}U$ age of 99.8 ± 3.3 Ma (2σ, MSWD = 1.7; n = 16; Fig. 1C). The same grains yield rare earth element and Ti abundances typical for crustal zircon and εHf values rangeing from 2.8 to −4.4 (n = 16; Item DR1). No evidence of inheritance was detected within analytical uncertainty.

Zircon $\delta^{18}O$ by SIMS

Measured $\delta^{18}O$ for individual 15 μm spots on zircon grains ranges from 13.8 ± 0.5‰ to 27.0 ± 0.7‰ (2σ), with a mean zircon $\delta^{18}O$ for the suite of 19.7 ± 2.9‰ (1σ, n = 125 analyses on 112 zircon grains; Figure 2; Items DR1, 2). Grains in most samples

record minor within-sample variation of $\delta^{18}O$ (<2‰), whereas in some samples grain-to-grain variability is up to 11‰. Variable $\delta^{18}O$ values also occur within individual grains (Fig. 3A). Multi-spot analysis shows that some grains are homogenous in $\delta^{18}O$, whereas others are zoned by up to ~9‰ (item DR1). The highest $\delta^{18}O$ zircon (Fig. 3A) was analyzed an additional 57 times using a ~3 μm spot (Fig. 3B), with $\delta^{18}O$ ranging from 17.7 ± 1.6‰ to 30.7 ± 1.5‰ in a concentric pattern. The ~3 μm spot analyses reveal a 28‰ core surrounded by a 19‰ rim (Fig. 3C). Concurrent analysis of $^{16}O^{1}H/^{16}O$ yielded low background-corrected values (0–0.0006), which showed no significant deviation during the 57 analyses, indicating the core is not ~~metamict (Wang et al.,~~ 2014see Item DR1).

Whole Rock and Quartz $\delta^{18}O$ by Laser Fluorination

Measured whole rock granite and quartz $\delta^{18}O$ values range from 14.3 to 19.3‰ and 19.9–22.1‰, respectively (Fig. 2B). The whole rock $\delta^{18}O$ values are uniformly high for igneous rocks, although some are lower than predicted based on zircon; the range of measured zircon $\delta^{18}O$ values for high-silica granites (~76 wt.% SiO_2) would be in equilibrium with whole rock $\delta^{18}O$ ranging from 16 to 29‰ (Valley et al., 2005). Some of the whole rock and quartz $\delta^{18}O$ values thus record high temperature equilibrium with zircon, whereas other reflect variable exchange during sub-solidus alteration.

DISCUSSION

High $\delta^{18}O$ S-type Granite in the Oman UAE Ophiolite

The Oman-UAE peraluminous granite dike suite described here (Fig. 2B) are the highest $\delta^{18}O$ igneous rocks known (Cavosie et al., 2011). The previously reported highest

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$\delta^{18}O$ values for igneous zircon (~14‰), quartz (~18‰), and whole rock (~16‰) are from ~1.2 billion year old granitoids of the Frontenac terrane in Ontario, Canada (Fig. 2B; Peck et al., 2004). Formation of the high $\delta^{18}O$ Frontenac granitoids is attributed to subduction and melting of continental material during accretionary orogeny. Mean values presented here for zircon (~20‰), quartz (~21‰), and whole rock (~17‰) are significantly higher than those found in Frontenac granitoids.

All available geochemical data for the granite dikes, including high $\delta^{18}O$, peraluminous character, normative corundum, the presence of aluminous minerals (garnet, muscovite and andalusite), and low Fe, Mg, Ca, and Na, identify the suite as S-type granite, having originated through melting of supracrustal materials (Chappell and White, 1974). Identification of crustal Nd and Hf isotope signatures (Haase et al., 2015) and elevated trace element abundance (Rollinson, 2015) in studies of peraluminous granite from the Oman-UAE ophiolite further support their classification as S-type granite.

Previous studies of zircon in S-type granite have found anomalously large intra-sample variation in igneous $\delta^{18}O$ (up to 5%), with ubiquitous inherited cores (e.g., Appleby et al., 2010). The S-type granites reported here show even greater intra-sample variation in zircon $\delta^{18}O$, from 14 to 28‰, including grains zoned by 9‰ (Fig. 3). The granites reported here are further distinguished from any other S-type granite in that the unimodal U-Pb ages preclude a detrital origin for the high $\delta^{18}O$ zircon; no evidence of inheritance was detected.

Oxygen Isotopes Constrain a Sediment Source

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We hypothesize that high $\delta^{18}O$ pelagic mud (pelitic and/or siliciceous) was subducted and melted below rapidly forming ocean crust during slab rollback (Rioux et al., 2013; Searle et al., 2015). The high $\delta^{18}O$ values measured for zircon, quartz, and whole rock uniquely restrict the melt source of the peraluminous granite suite to high $\delta^{18}O$ sediment. The extremely elevated $\delta^{18}O$ signatures for igneous zircon exclude a mantle source, as whole rock values for peridotite and derivative melts do not exceed ~6‰ (Valley et al., 2005). A global survey of whole rock values identifies shale ($\delta^{18}O$ up to 26‰; Payne et al., 2015) as the most likely high $\delta^{18}O$ source that would yield peraluminous granite when melted (Nichols et al., 1994). Other possible high $\delta^{18}O$ sources include siliceous and carbonate oozes, which have whole rock $\delta^{18}O$ values from 25 to 42‰ (Eiler, 200). However, melts of oozes or lithified equivalents would not form peraluminous granite, which limits their contributions, if present. Linear mixing of equal parts altered basalt (50 wt% SiO_2, $\delta^{18}O$ = ~9‰) and chert (100 wt% SiO_2, $\delta^{18}O$ = ~40‰) could produce a $\delta^{18}O$ melt (~21‰) in equilibrium with the mean zircon $\delta^{18}O$ (~19‰, Fig. 2), however SiO_2 would be too low (70 wt. %), and equilibrium with the highest zircon

$\delta^{18}O$ value (Fig. 3) is not attainable. More reasonable melt components include shale (60 wt% SiO_2, $\delta^{18}O$ = ~20‰) mixed with 30%–40% chert, which could produce a melt with ~75 wt% SiO_2 and whole rock $\delta^{18}O$ of up to ~28‰. Regardless of lithologies chosen for modeling or extent of melting (partial versus bulk), unusually high $\delta^{18}O$ sources are required to crystallize the ~28 to 19‰ zircon domains. The zircon core-to-rim

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relationships indicate the source was clearly heterogeneous, resulting in melt $\delta^{18}O$ changing evolved over time, either in the dynamic environment of the subduction channel or in the overlying mantle wedge.

No appreciable volume of shale is exposed near the Oman-UAE ophiolite on the paleo-continental shelf (Searle et al., 2015; Searle and Cox, 1999). Siliceous schist, marble, and chert, all with high whole rock $\delta^{18}O$, occur regionally (Grantham et al. 2003), however these lithologies do not have an appropriate composition to form S-type granite when melted (Rollinson, 2015). The lack of shale in the region is not surprising, as the majority of pelagic pelitic mud in modern marine settings is found further offshore and would have been among the first continentally-derived material subducted at ~99 Ma. Pelagic zircon-free sediment from the Arabian continent is consistent with εHf values and lack of age inheritance (Fig. 1C; Item DR1).

Melting Mud in the Mantle

Data presented here from the Oman-UAE ophiolite provide direct geochemical evidence for melting of subducted sediment within the mantle from an intact section of mantle lithosphere. Prior estimates for sediment components in the Oman-UAE mantle granite suites range from 10%–30% (trace elements, Rollinson, 2015), to near 80% (Hf-Nd isotopes, Haase et al., 2015), however our results constitute the first report of pure sediment melting. Oxygen isotopes are a highly sensitive tracer of melt source; no indication of mantle-derived melt was detected in the peraluminous granite suite, nor is one required, as the granite compositions can be fully explained by melting of high $\delta^{18}O$ marine sediment. The peraluminous granite suite described here may thus represent the

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first report of S-type granite that contains no detectable component of 'primitive melt' (c.f. Kemp et al., 2008).

Recognition of the peraluminous granite dikes as nearly pure sediment melts may lead to new insights into related subduction zone processes, including constraining the steady-state volume of subducted sediment needed to source the melts, and the rheology and thermal state of the mantle wedge to allow for semi-brittle fracture formation and propagation during dike emplacement. The initiation of subduction, formation of oceanic crust, melting of sedimentary material, and intrusion of S-type granite into the mantle wedge all pre-date thrusting of the oceanic crust onto the Arabian continental margin to form the Oman-UAE ophiolite (Searle et al., 2015; Searle and Cox, 1999). These data thus provide "ground truth" confirmation for sediment subduction and melting beneath supra-subduction zone ophiolites (Furnes and Dilek, 2017). The recognition that small-volume, isotopically evolved, granitoid reservoirs form in the upper mantle as a consequence of subduction also has implications for the compositional heterogeneity of the mantle over time.

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FIGURE CAPTIONS

Figure 1. A: Simplified map of the Oman-UAE ophiolite (Rollinson, 2015) with locations of sampling sites. B: Photograph of peraluminous granite intruding mantle peridotite (harzburgite) near Al-Dadnah, UAE. C: Terra-Wasserberg concordia of U-Pb data for granite samples from this study.

Figure 2. A: Histogram of $\delta^{18}O$ zircon analyses (15 μm SIMS spots) from peraluminous granite in this study compared with global $\delta^{18}O$ compilations of igneous (Cavosie et al., 2011) and detrital (Spencer et al., 2014) zircon data. Note that 99.8% of detrital and 100% of igneous grains are below 13.8‰, the lowest measured $\delta^{18}O$ values of zircon from the peraluminous granite reported here. B: Zircon, quartz, and whole rock $\delta^{18}O$ values from Frontenac terrane granitoids (Peck et al., 2004) and peraluminous granite (this study).

Figure 3. A: Cathodoluminescence (CL) image of zircon UAE436_1, displaying locations of the three 15 μm $\delta^{18}O$ spots. Faint growth zoning in the dark core parallels zoning in rim, indicating primary magmatic growth rather than inheritance. B: Backscatter electron image showing the fifty-seven ~3 μm spot analyses for $\delta^{18}O$. C: CL image of the same surface shown in B with color-coded analytical spots based upon the color scale. The spot with a cross in C is excluded, as it overlapped an inclusion visible in B. A histogram and kernel density estimation (dashed curve) are shown right of the color scale (bin- and bandwidth = 1.5‰). Background corrected $^{16}O^{1}H/^{16}O$ (using $\delta^{18}O$ standard 91500) is shown as a function of $\delta^{18}O$ (vertical line) and is nearly invariant. All $\delta^{18}O$ values are in ‰, with average uncertainties shown in A and B.

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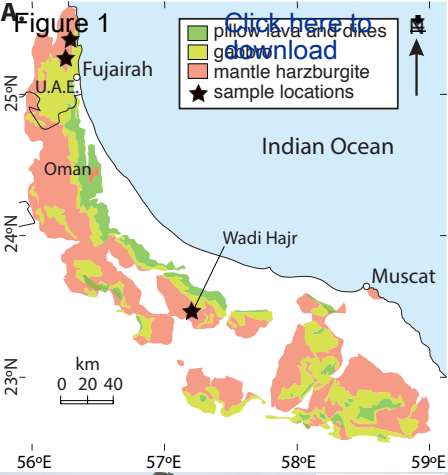
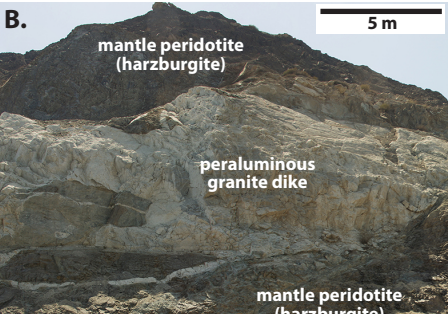
1GSA Data Repository item 2017xxx, material, methods, and full data tables, is available online at http://www.geosociety.org/datarepository/2017/ or on request from editing@geosociety.org.

Figure 1





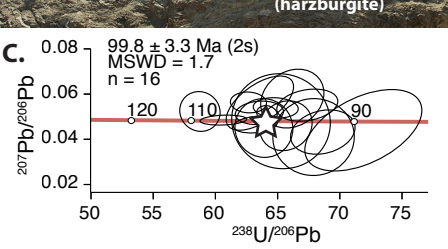


Figure 2

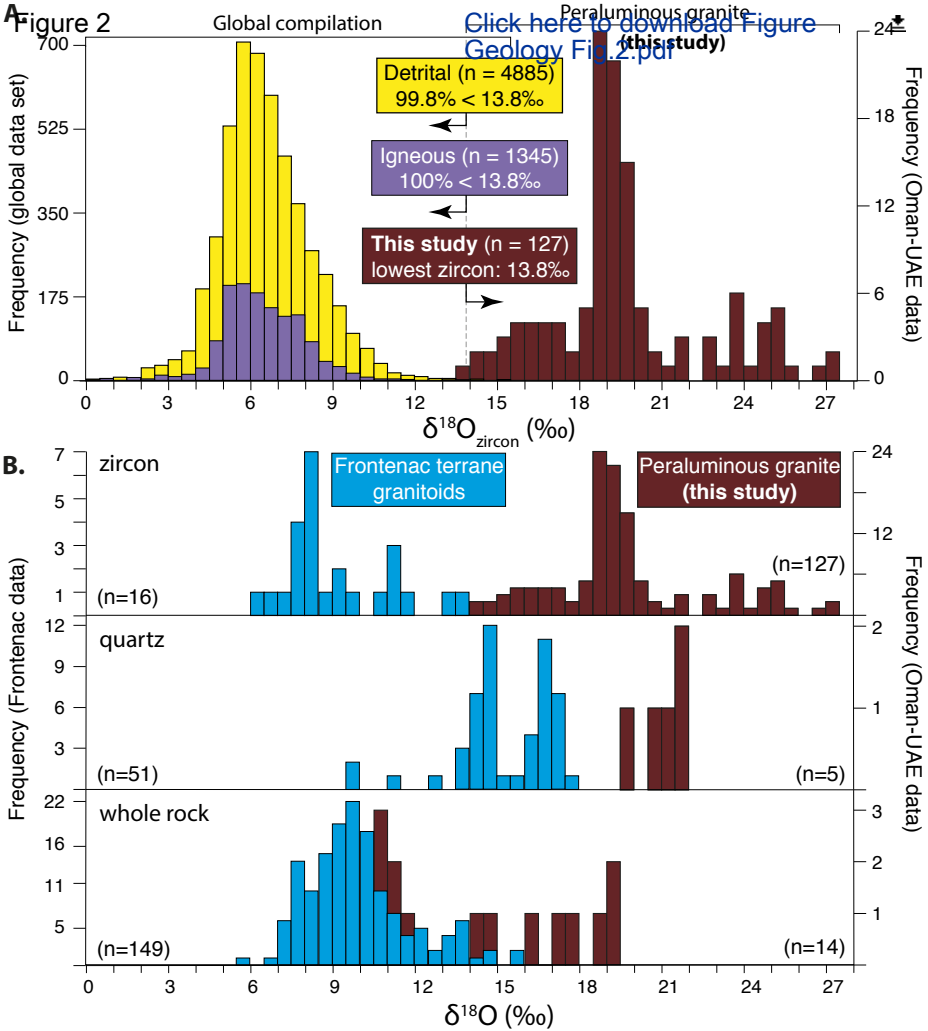

A.
Global compilation
Peraluminous granite
(this study)
Click here to download Figure Geology Fig.2.pdf
Detrital (n = 4885)
99.8% < 13.8‰
Igneous (n = 1345)
100% < 13.8‰
This study (n = 127)
lowest zircon: 13.8‰
Frequency (global data set)
Frequency (Oman-UAE data)
δ18Ozircon (‰)
B.
zircon
Frontenac terrane granitoids
Peraluminous granite (this study)
(n=16)
(n=127)
quartz
(n=51)
(n=5)
whole rock
(n=149)
(n=14)
Frequency (Frontenac data)
Frequency (Oman-UAE data)
δ18O (‰)

Figure 3